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Asphaltene Solvency and Stability of Water in Oil Emulsion: A Case Study of Two Nigerian Crudes

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Abstract

The effect of asphaltene solvency on the stability of water-in-oil emulsion was investigated using crude samples from two Nigerian oil wells, namely Okoro oil well (crude A) and Okpoho oil well (crude B), chosen because of their varying resin and asphaltene content. Solvency of the emulsion was modified by addition of heptane, toluene, and their blend (heptol) in various proportions. The effect of aqueous phase pH on the stability of the emulsion was also studied. The results showed that the Okoro well crude oil which has higher asphaltene content of 3.2 wt% and lower resin/asphaltene ratio of 1.15 formed a much more stable emulsion than that of Okpoho oil well which has 1.9 wt% asphaltene content and 3.16 resin/asphaltene ratio. It was also noticed that the emulsion became more destabilized at basic pH range. A model was developed which can be used to predict the stability of the water/oil emulsion in period of time beyond the study range.

Keywords: water-in-oil emulsion, stability, asphaltene, resin, solvency, aromaticity

1. Introduction

Emulsions consist of a dispersion of an immiscible liquid (dispersed phase) in another liquid (continuous phase) with drop size usually in the micrometer range. They are generally classified in three categories of water-in-oil (W/O), oil-in-water (O/W), and complex (multiple) emulsions (W/O/W or O/W/O) (Hoshyargar and Ashrafizadeh, 2013; Roudsari et al., 2012; Sjoblom, 2001). Crude oil commonly exists in the form of water-in-oil emulsions. These emulsions are formed during the production of crude oil, which is often accompanied by water. Natural surfactants such as asphaltenes, resins and carboxylic acid, and solids such as clays and waxes stabilize these emulsions. The emulsions have stability ranging from a few minutes to years, depending on the nature of the crude oil and the extent of water. It is essential to break these emulsions before transportation and refining (Al Sabagh et al., 2008; Djuve et al., 2001; Joseph and Peter, 1997; Mosayebi and Abedini, 2013). However, treatment of these water-in-oil emulsions is still a challenge in the petroleum industry due to their high stability versus coalescence owing to substances, such as asphaltenes and resins, with polar characteristics forming resistant films at the oil-water interface (Ortiz et al., 2010; Aguilar et al., 2013; Kokal, 2005; Speight, 2004).

Asphaltenes are high molecular weight solids which are soluble in aromatic solvents such as benzene and toluene and insoluble in paraffinic solvents (Ashoori et al., 2010; Speight et al., 1985, Abedini et al., 2011; Eskin et al., 2011). Resins are insoluble in ethyl acetate and soluble in aliphatic hydrocarbons with low molar mass such as n-heptane and in aromatic solvents such as benzene and toluene (Aguilar et al., 2013). Both asphaltenes and resins correspond to the heavy fraction of crude oil composed of polar molecules. Their structure contains heteroatoms such as nitrogen, oxygen, and sulphur, and metals such as nickel, vanadium, and iron. However, asphaltenes have higher molar mass, aromaticity, greater quantity of heteroatoms and metals (Aguilar et al., 2013; Mullins et al., 2003).

Several studies have identified the predominant role of asphaltene and its state in emulsion stability (Ali and Alqam, 2000; Aske et al., 2002; Kokal, 2005; Mouraille et al., 1998; Sjoblom et al., 2001; Jestin et al., 2007; Ortiz et al., 2010; Dicharry et al., 2006; Xia et al., 2004; Verruto and Kilpatrick, 2008). Their stabilizing properties are significantly enhanced when they precipitate and aggregate (Aguilar et al., 2013).

Ali and Alqam (2000) investigated the various factors affecting the stability of w/o emulsions in some crude oil samples from Eastern Province of Saudi Arabia. They proposed that resins increase the stability of asphaltenes in the crude and hence minimize the asphaltene interaction with water droplets. Hence, the resins – asphaltenes ratio decreases as emulsion become tighter and harder to break.

McLean and Kilpatrick (1997) using four crude samples, namely Arab Berri (extra light), Arab Heavy, Alaska North slope and San Joanquin valley, analyzed the influence of the solvency of asphaltenes on the stabilization of emulsions. They noted that asphaltenes act to stabilize w/o emulsions when they are at or near the precipitation point. The results showed a reduction in the stabilization of the emulsions tested when the solvency degree of the asphaltenes was altered from the aggregate state to the molecular state. Asphaltene aggregates are adsorbed at the water-oil interface by the hydrogen bonds or other interactions between the water and polar portions of the aggregates.

Xu et al., (2013) investigated the effect of water content and temperature on the stability of w/o emulsion using samples from Jilin oilfield. Their results indicated that emulsion stability decreases when water content or holding temperature increases.

To the best of our knowledge there has been no reported study of w/o emulsion with respect to any Nigerian crude oil. Yet it is well known that no two crudes exhibit similar properties and crude oil emulsions defy broad and generic resolution (McClean and Kilpatrick, 1997; Abedini et al., 2011). In this work, we investigate the effect of asphaltene solvency on the stability of water-in-oil emulsion using two Nigerian crudes, namely Okoro well crude (crude A) and Okpoho well crude (crude B). Solvency was influenced by paraffinic solvent (n-heptane), aromatic solvent (toluene), a blend of both (heptol) in various proportions, and pH of aqueous phase.

2. Materials and Method

2.1 Materials

Crude oil samples were collected from two different oilfield wells in Nigeria, namely Okoro well in Akwa Ibom State waters operated by Afren Energy (denoted Crude A) and Okpoho well in Bayelsa State operated by shell petroleum Development Company (denoted Crude B). The chemicals used includes n-heptane, sodium hydroxide pellet, hydrochloric acid and toluene were all of analytical grade.

2.2 Methods

SARA characterization of the crude samples was carried out together with density and viscosity measurement as presented in Table 1. The amount of resolved water was considered the most appropriate measure of the emulsion stability of water-in-oil emulsions, since the coalescence of the droplet phase is the limiting step in the demulsification process (Xia et al., 2004).

Table 1: Physico-chemical properties for the two crude oils.

Crude oil properties	Okoro well crude oil (crude A)	Okpoho well crude oil (crude B)
Saturates, weight %	64.80	47.19
Aromatics, weight %	28.30	44.91
Resins, weight %	3.7	6.0
Asphaltenes, weight %	3.2	1.9
Resins/Asphaltenes ratio	1.15	3.16
Density, kg/m^3	865.4	822.0
Viscosity, mPa.s	4.8082	1.4504

2.3 Emulsion preparation and monitoring process

To ensure homogeneity of the crude oil samples, the whole crude were mixed thoroughly by a vigorous hand shaking. The emulsion was prepared by adding 20 ml of the oil sample into a 400 ml polypropylene jar and 60 ml of water with pH value 7.07. The crude was mixed with Silverson SL2 high speed laboratory mixer at 4000 rpm while the water was added in drops into the jar. After the addition of the 60 ml water to the oil sample in the jar, the speed of the mixer was adjusted to 7000 rpm for 5 min in order to achieve a good emulsion. The emulsion was then transferred into a 100 ml cylinder and allowed to settle under gravity for 24 hr. A 2 hr interval determination of the amount of water resolved by the emulsion was taken and recorded. The emulsions were prepared at 20 ^{o}C and atmospheric pressure and allowed to settle under gravity at room temperature and atmospheric pressure.

The same procedure was carried out to monitor the effects on the emulsion stability by the addition of surface active solvents (n-heptane, toluene, and Heptol) and change in pH of the aqueous phase (water). The solvents n-heptane, toluene and heptol 50 (50% n-heptane and 50% toluene) were added to the crude from 10 % - 50 % of the crude oil volume. The mixture was mixed using the Silverson SL2 laboratory mixer at 2000 rpm for 2 min to ensure proper mixing. The emulsion preparation process as stated above was then carried out. Change in pH value of the aqueous phase was carried out by adjusting the pH of the water using hydrochloric acid (HCl) and Sodium hydroxide (NaOH). The pH ranges of 2.01, 4.00, 7.07, 9.45 and 12.00 were used to analyse the effect of pH change on the stability of the emulsion.

4.0 Results and discussion

The emulsion stability results for the two crude oils in this study which was monitored under gravity sedimentation are presented as a function of time and crude type in Fig.1. It should be noted that the emulsion stability increases as the amount of water resolved decreases.

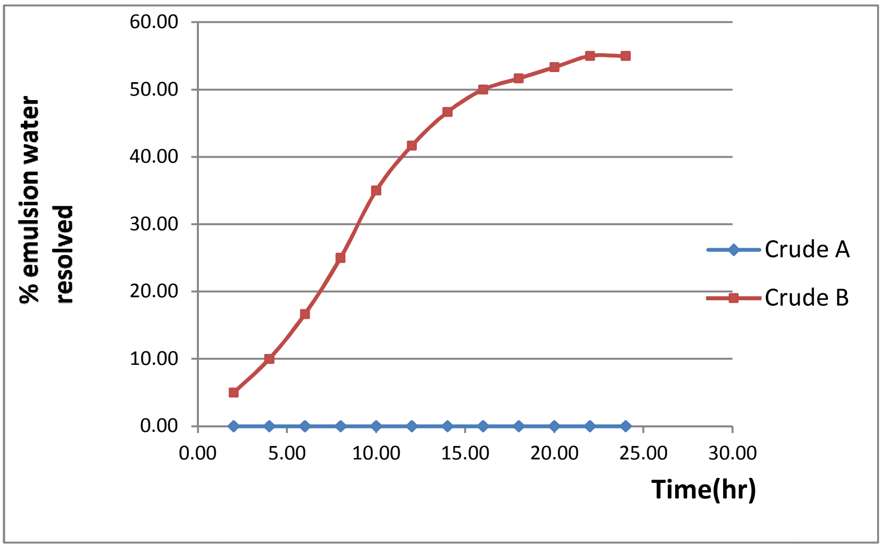

Fig.1: Effect of gravitational settling on the emulsions stability

It is apparent that Crude A formed a very stable emulsion and did not resolve any water on gravitational settling for 24 hr while Crude B resolved 55 % of the emulsion water under gravity settling for 24 hr. This is due to their differences in resin to asphaltene ratio and aromatic content of the crudes. Crude B has a resin to asphaltene ratio of (R/A) of 3.16 and an aromatic content of 44.91 while Crude A has R/A of 1.15 and an aromatic content of 28.30. From literature, crude oils with high resin to asphaltene ratio tend to form unstable water in oil emulsion. This is attributed to the fact that the asphaltenes are highly solvated and forms a weak interfacial film between the water droplets and the oil and has a low tendency to stabilize the emulsion (McLean and Kilpatrick, 1997; Xia et al., 2004; Al-Sabagh et al., 2011). Also, the presence of high aromatic compound in the crude oil tends to solvate the asphaltenes more which reduces their ability to adhere to the water/oil interface and thereby forms a less stable water/oil emulsion (McLean and Kilpatrick, 1997).

3.1 Effect of addition of solvents on the emulsion stability

It is known that asphaltenes solvency is responsible for emulsion stability in water and crude oil emulsion. This study is aimed to determine the effect of changing the nature of the crude medium by blending the crude with solvents of varying amount of aromaticity. Here, the crude oil was modified by the addition of a purely aliphatic solvent (n-heptane), an aromatic solvent (toluene) and a mixture of the two (heptol; 50% heptane and 50% toluene) in increasing quantity of 10 – 50 %.

3.1.1 Effect of n-heptane addition

The result showed that crude A did not resolve any water in all the fraction of n-heptane addition. Also a remarkable situation was observed as the emulsion formed becomes more viscous as the percentage addition of n-heptane added increases. Emulsion with the 40% and 50% n-heptane addition was highly viscous and looks like a semi solid. This is attributable to the fact that asphaltenes are insoluble in n-heptane and when n-heptane is added in a small fraction to the crude oil, it will not be able to precipitate the asphaltenes out of the crude oil but will alter the nature of the solubility of asphaltenes in the crude oil by making the asphaltenes partially solvated (state of dispersion). From literature, crude oil forms more stable emulsion with water when the asphaltenes are in a state of dispersion rather than when they are in a state of dissolution (McLean and Kilpatrick, 1997). For Crude B, it was noticed that the stability of the water in oil emulsion formed as a result of added n- heptane increases as the fraction of n-heptane added increases as shown in Fig. 2.

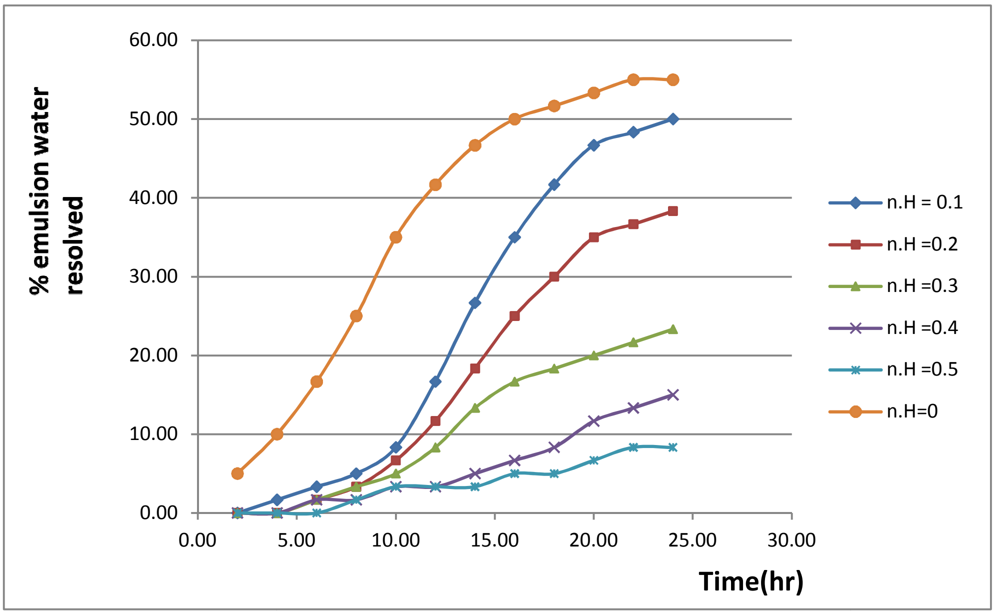

Fig. 2: Effect of n-heptane addition in Crude B sample to emulsion stability.

The result indicates that the percentage water resolved reduced from 55% when there is no addition of solvent to 8.33% for the addition of 50% n-heptane. This can be seen clearly in Fig. 3 which is a representation of the effect of n-heptane addition on the two crude oils at 24hr.

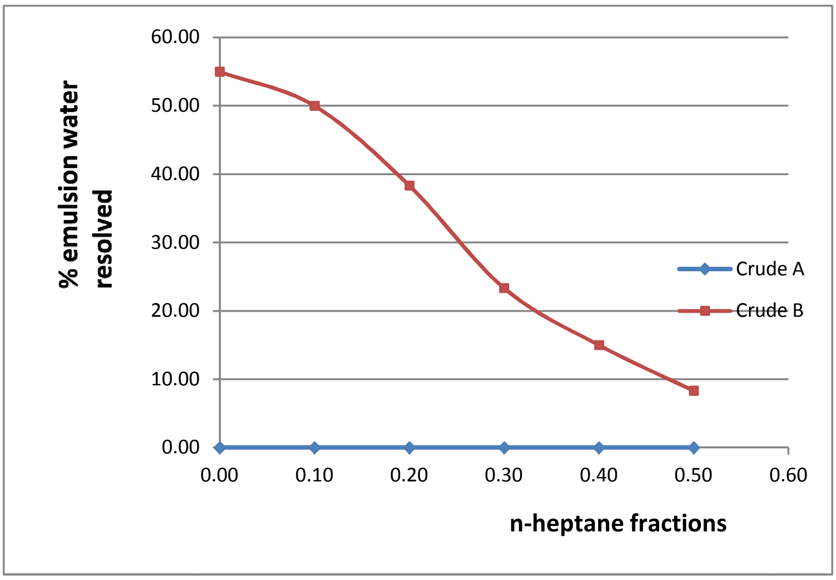

Fig.3: Effect of n-heptane addition on the two crude oil samples at 24 hr

3.1.2 Effect of toluene addition

Fig. 4 shows that crude A resolved some part of its emulsion water on addition of toluene. The process monitored with time showed that crude A did not resolve any water at 0 and 10 % addition of toluene, only 1.67 % of water was resolved at 20 % addition of toluene, 5 % water content was resolved both in the addition of 30 and 40 % toluene while 10 %emulsion water was resolved at 50 % addition of toluene.

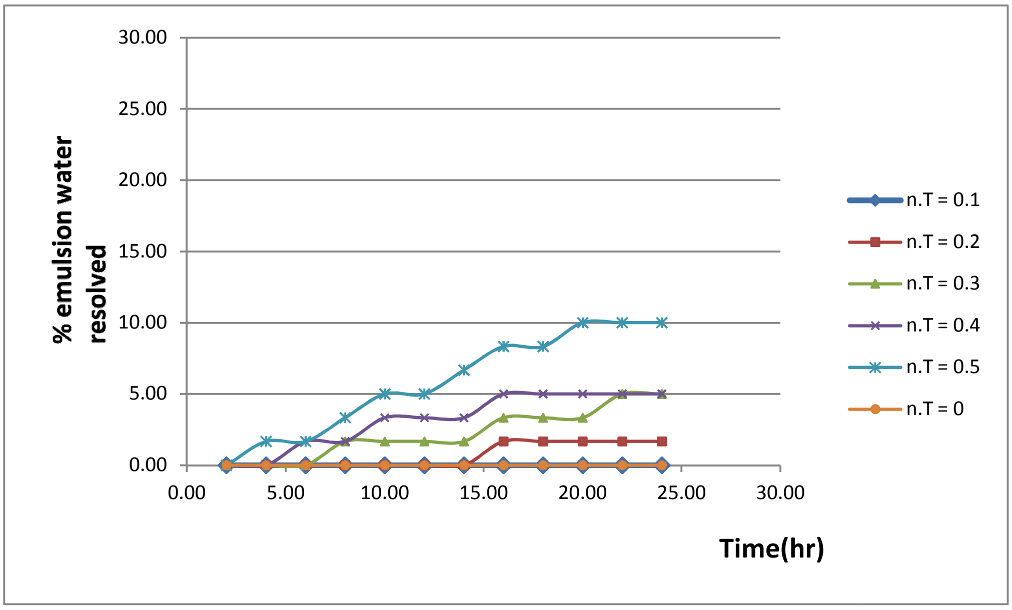

Fig.4: Effect of toluene addition to Crude A sample on emulsion stability.

Crude B proved to be much unstable to addition of toluene as shown in Fig.5, there was progressive increase in the emulsion water resolved with increase in the percentage of added toluene. It shows that the emulsion becomes more unstable as the toluene fraction added to the crude oil increases as was the case with crude A.

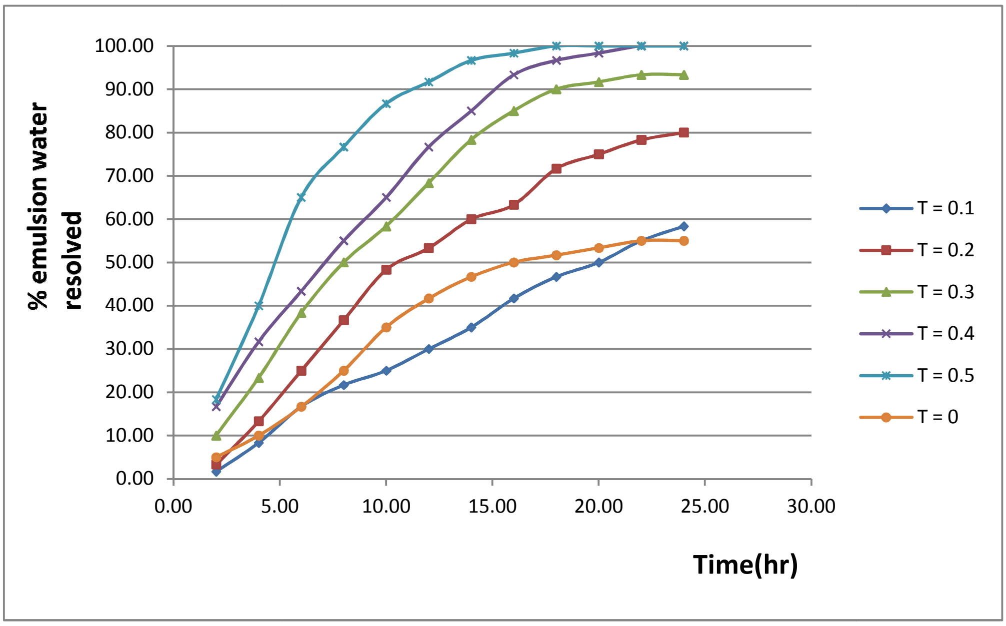

Fig.5: Effect of toluene addition to Crude B sample on emulsion stability.

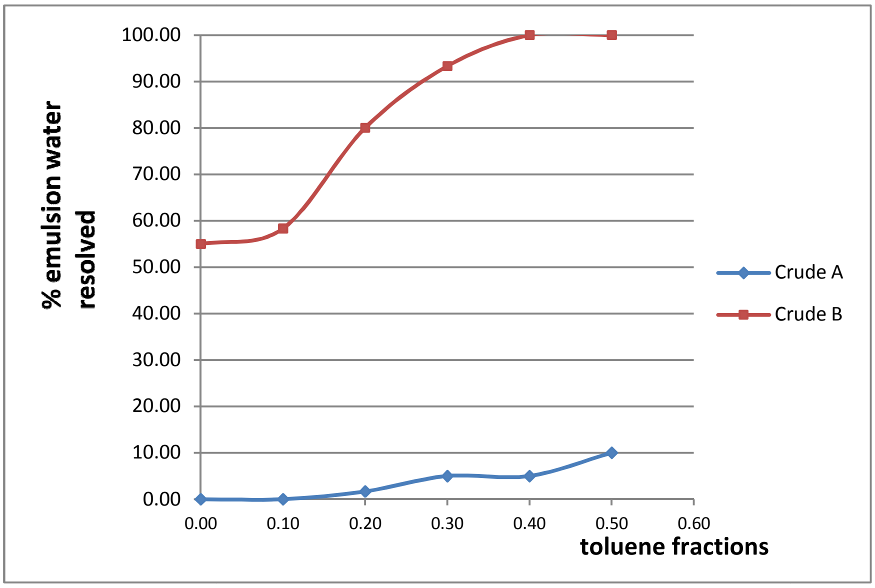

Fig.6: Effect of toluene addition on the two crude oil samples at 24 hr

This indicates that the toluene further solubilise the asphaltenes in the crude oil and reduces its tendency to form a stable emulsion with the water droplet. When asphaltenes are highly solubilised, they form a weak interfacial film between the water droplets and the oil medium (Jones et al, 1978 and McLean and Kilpatrick, 1997). These films will rupture quickly with time to liberate the water which now settles down at the bottom of the cylinder.

3.1.3 Effect of heptol (50% n-heptane ,50% toluene.) addition

Crude A formed a stable emulsion on addition of the heptol fractions to the crude oil samples. No water was resolved throughout the heptol fractions but there was dense clustering of water droplets at the bottom of the cylinder for the 40 and 50% addition of heptol to the crude oil.

Crude B showed a remarkable resolution of the emulsion water with addition of heptol fractions as shown in Fig.7.

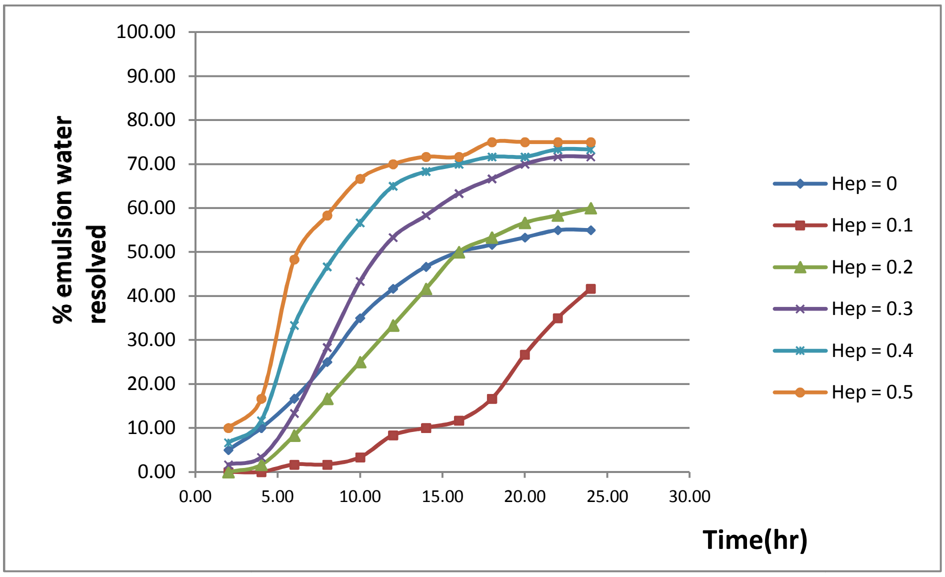

Fig.7: Effect of heptol addition to Crude B sample on emulsion stability.

The percentage water resolved increases with increase in the fraction of heptol added to the crude oil sample. It was noticed that the addition of 10 % heptol resolve less water than when there is no addition of any solvent in the crude oil sample. This can be attributed to the more influence of n-heptane on the crude oil sample than toluene at a lower concentration addition of the mixture of the two solvents to the crude oil. The 20 % addition of heptol sample initially resolved lower quantity of water than the 0 % sample but later surpass that of 0 % just at 18 hr of settling. Also, the 10-30 % heptol added samples had an initial low start with water resolution than that of 0 %, which also show the effect of the n-heptane present in the heptol mixture. But the addition of heptol on higher fractions like 40 and 50 % showed remarkable resolution of the emulsion water and though that of 30 % addition had an initial low water resolution characteristics but it later resolved a larger percentage of the emulsified water that is 71.66 %. This can be well seen in Fig. 8 which shows the percentage water resolved with respect to added heptol fractions at 24 hr.

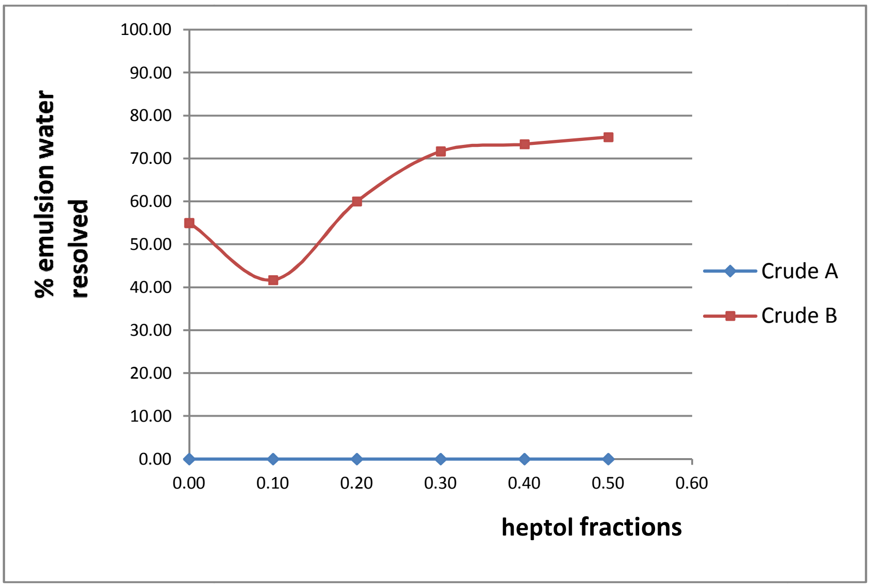

Fig. 8: Effect of heptol addition on the two crude oil samples at 24 hr

In general, the influence of addition of the three solvents to the crudes can be analysed. In crude A, it was discovered that it only resolved its emulsified water due to addition of toluene with a maximum of 10% water content resolved. This is as shown below in Fig. 9.

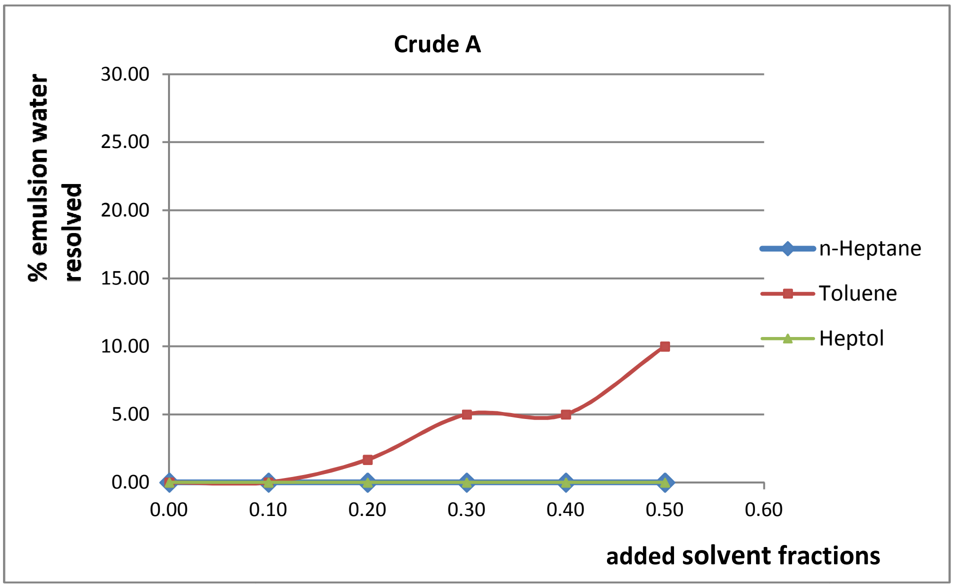

Fig. 9: Effect of added solvents on crude A sample at 24 hr

This shows that crude A has a higher tendency to form stable emulsion with water even when there are changes in the solvency of the asphaltenes and resins in the crude. This can be attributed to its low resin/asphaltene ratio of 1.15 and a bit high asphaltenes content of 3.2 wt%.

On the other hand, crude B proved to be totally unstable due to addition of toluene, it gave away all the water used in the preparation of the emulsion at both the addition of 40% and 50% of toluene to the crude oil sample. This is shown below in Fig. 10.

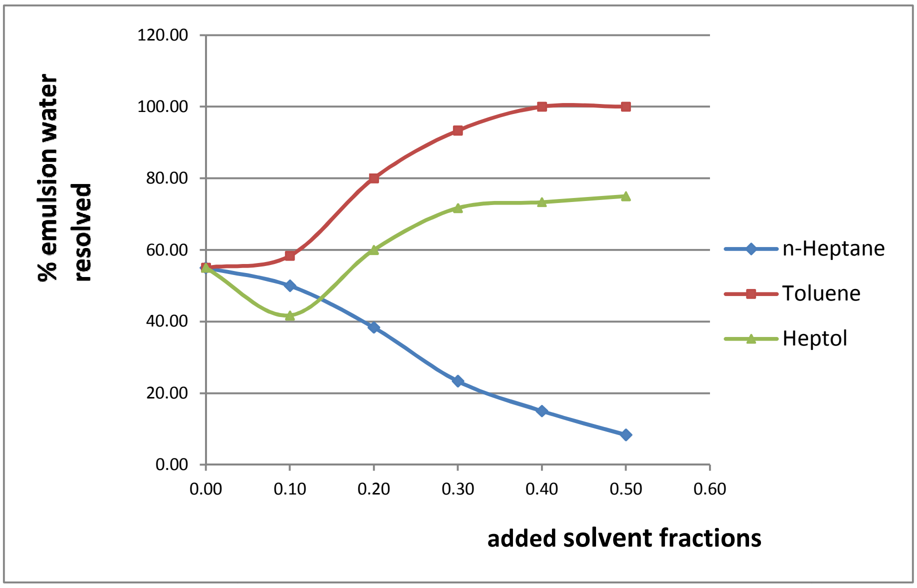

Fig. 10: Effect of added solvents on crude B sample at 24 hr

It also gave away large amount of the emulsified water due to addition of heptol with a maximum of 76.66 % on addition of 50 % heptol to the crude oil sample. This can be attributed to the high value of the resin/asphaltene ratio of 3.16, low asphaltene content of 1.9 % and high aromatic content of 44.91 %. But it showed a remarked tendency towards stability by the addition of n-heptane by resolving only 10 % of the emulsified water on addition of 50 % n-heptane to the crude oil sample.

3.2 Effect of pH change of the aqueous phase (water)

Asphaltenes and resin molecules contain both acidic and basic components. So change in pH of the aqueous phase (water) will affect the emulsion stability of the water in crude oil emulsion (McLean and Kilpatrick, 1997; Sjoblom et al., 1992; Hashmi and Firoozabadi, 2013). This was experienced in this work as the influence of pH on the stability of w/o emulsion was studied with the two crude oil samples. Fig.11 shows the effect of varying the pH of the aqueous phase used in forming emulsion with crude A.

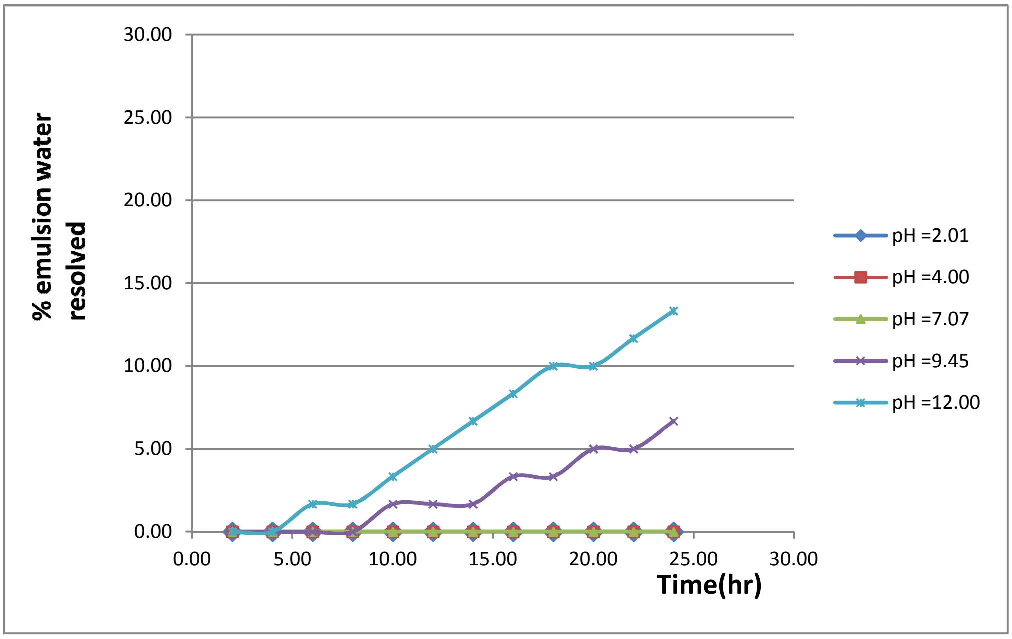

Fig. 11: Effect of pH change of the aqueous phase on the emulsion stability of Crude A sample.

It was noticed that crude A was very stable on the acidic media, just like in the addition of n-heptane to crude A, pH values of 2.01 and 4 produced highly viscous emulsion with no water resolved. That of 7.07 produced a good emulsion but yet no water resolved after 24 hr of settling under gravity. The higher pH values of 9.45 and 12 show water resolution with that at pH of 12 resolving the highest amount of water (13.33%) while that of 9.45 resolved 6.66% of the emulsified water. Crude B also showed a similar trend to that of crude A as it formed a more stable emulsion with the acidic range and became very unstable in the basic range. This is shown in Fig. 12.

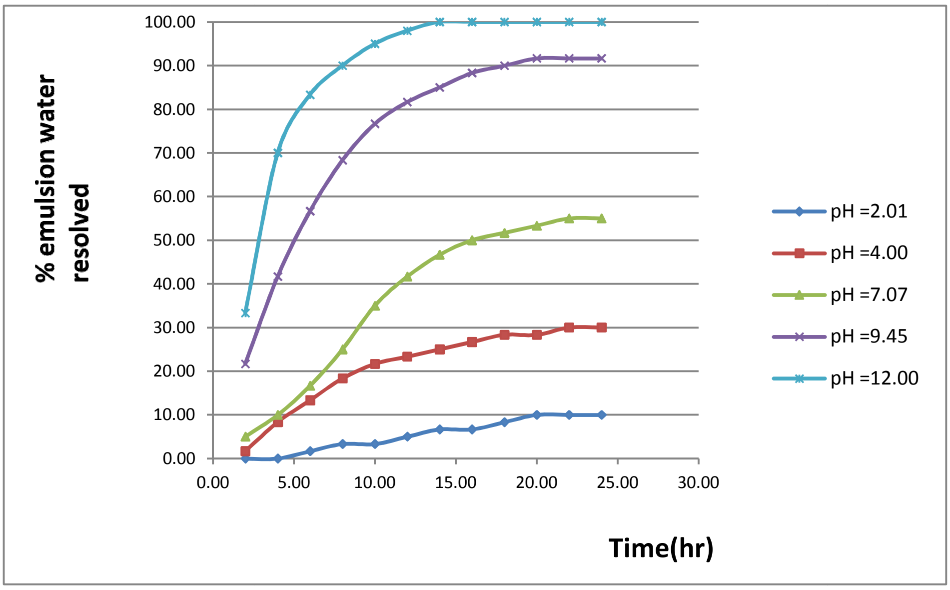

Fig. 12: Effect of pH change of the aqueous phase on the emulsion stability of Crude B sample.

The emulsion was very unstable to high basic pH of 12 and gave away all the emulsified water at 14hr of gravitational settling but formed more stable emulsion with the acidic range of pH 2 and 4. So as the pH of the aqueous phase increases, the stability of the emulsion formed decreases. Increasing pH causes changes in film thickness and asphaltene concentration due to the ionization of acidic components of asphaltene on account of deprotonation at basic pH (Jestin et al., 2007; Verruto and Kilpatrick, 2008; Hashmi and Firoozabadi, 2013).

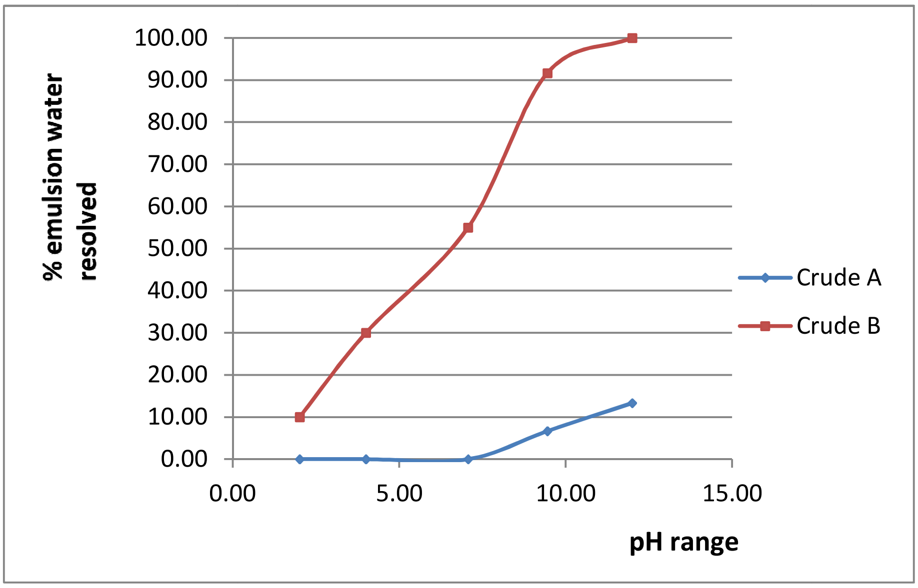

Fig. 13: Effect of pH change on the emulsion stability of the two crude oil samples at 24hr.

3.3 Modelling

The process of emulsion coalescence is a first other process defined by

$$A = D + W \quad 1$$

Where

A is initial emulsion volume, D represents final emulsion volume and W the volume of the final free water phase (Wanli et al, 2000).

This is applicable to the study of the stability rate of an emulsion by monitoring the retention of the emulsified water with time where,

$A = R + S$ 2

Where; A = initial water content of the emulsion
R = water retained by the emulsion at any time t.
S = water separated or resolved by the emulsion.

The rate at which the emulsion water diminishes can be define with a first order rate equation as

$(-r_A) = \frac{-dC_A}{dt} = k_c C_A$ 3

Where

$C_A = concentration\ of\ the\ water\ in\ the\ emulsion\ at\ any\ time\ t.$
$k_c = breakage\ rate\ constant\ of\ the\ emulsion.$
t = time (hr).

Therefore,

$(-r_A) = \frac{-dC_A}{dt} = k_c C_A$ 4

$$\int_{C_{Ao}}^{C_A} \frac{-dC_A}{C_A} = k_c \int_0^t dt$$

Integrating the equation above, we have

$\ln\left[\frac{C_{Ao}}{C_A}\right] = k_c t$ 5

The concentrations of the emulsion water is evaluated as the volume fraction of the water to that of the emulsion as

$C_{Ao} = \frac{V_{wi}}{V_e}\ and\ C_A = \frac{V_w}{V_{re}}$ 6

Where

$V_{wi} = initial\ water\ volume\ in\ the\ emuslion.$
$V_e = initial\ emulsion\ volume.$
$V_w = water\ volume\ in\ the\ emulsion\ at\ any\ time\ t.$
$V_{re} = remaining\ emulsion\ volume\ at\ any\ time\ t.$

Plotting $\ln\left[\frac{C_{Ao}}{C_A}\right]$ against t will give a straight like with k_c as the slope of the graph as shown below in Fig 14.

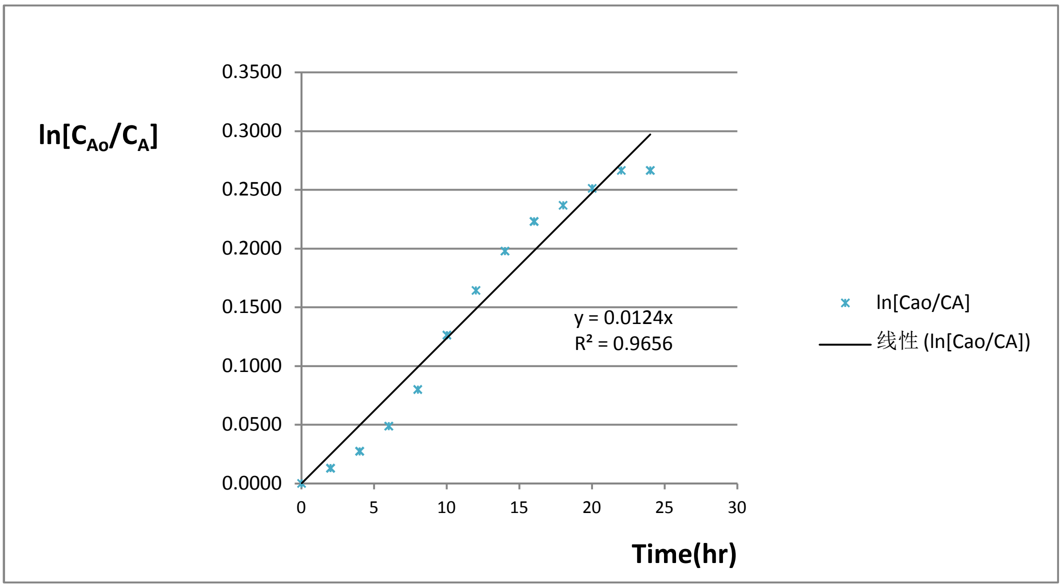

Fig. 14: A plot of $\ln\left[\frac{C_{Ao}}{C_A}\right]$ against t for Crude B.

From the graph, the slope of the graph k_c which is the breakage rate constant of the emulsion is evaluated as $k_c = 0.0124hr^{-1}$.

The individual calculated rate for every two hours was monitored and compared to the experiment rates for every two hours interval as shown in Fig. 10., where

Calculate rate is $(-r_A)_{cal} = k_c C_A$ and

The experimental rate is $(-r_A)_{exp} = \frac{-dC_A}{dt} = -\left[\frac{C_{Ao}-C_A}{t_o-t}\right]$

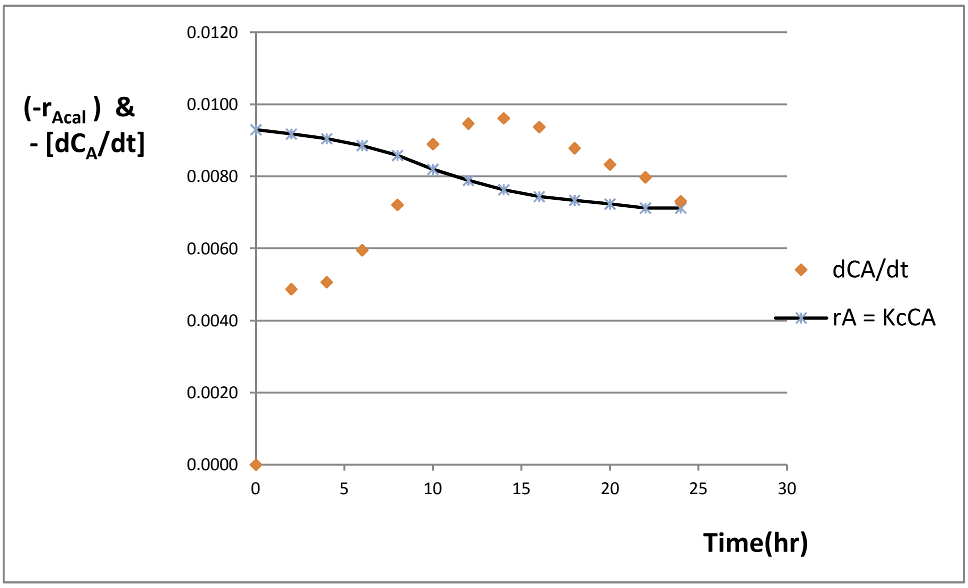

Fig. 15: A plot of $(-r_A)_{cal}$ and $\frac{-dC_A}{dt}$ against t.

The experimental rates showed that the rate of water resolution steadily increase with time till it reached a maximum at 14 hr and then reduces with time. The calculated rates showed a steady decrease with time and are attributed to the steady reduction in concentration of the emulsified water with time. It can be used to predict further state of the emulsion at any time. A comparison of the stability of the two crude oil samples based on their breakage rate constant is carried out using the data of 50% addition of toluene in the crude oil samples as shown below in Fig. 16a and Fig. 16b.

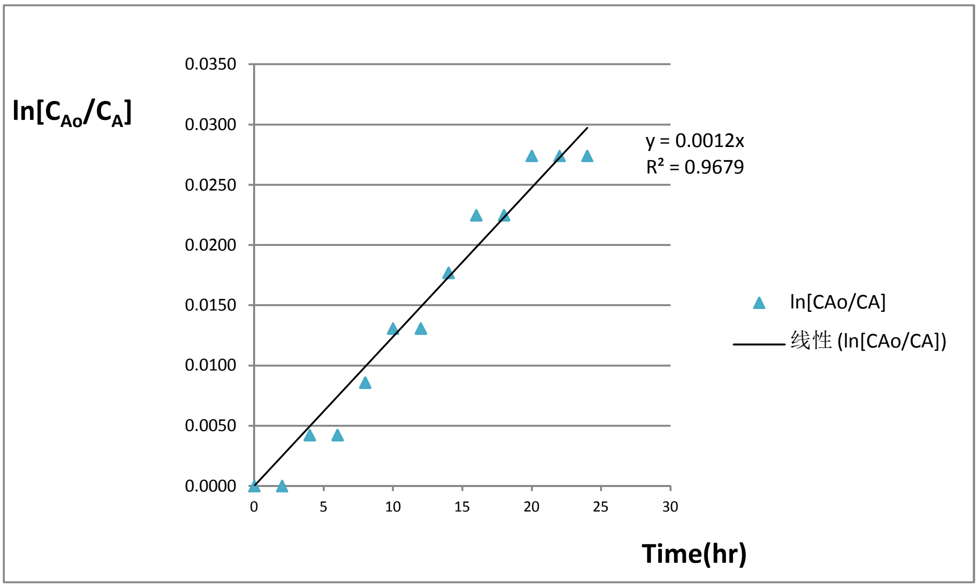

Fig.16a: A plot of $\ln\left[\frac{C_{Ao}}{C_A}\right]$ against t for crude A at 50% addition of toluene.

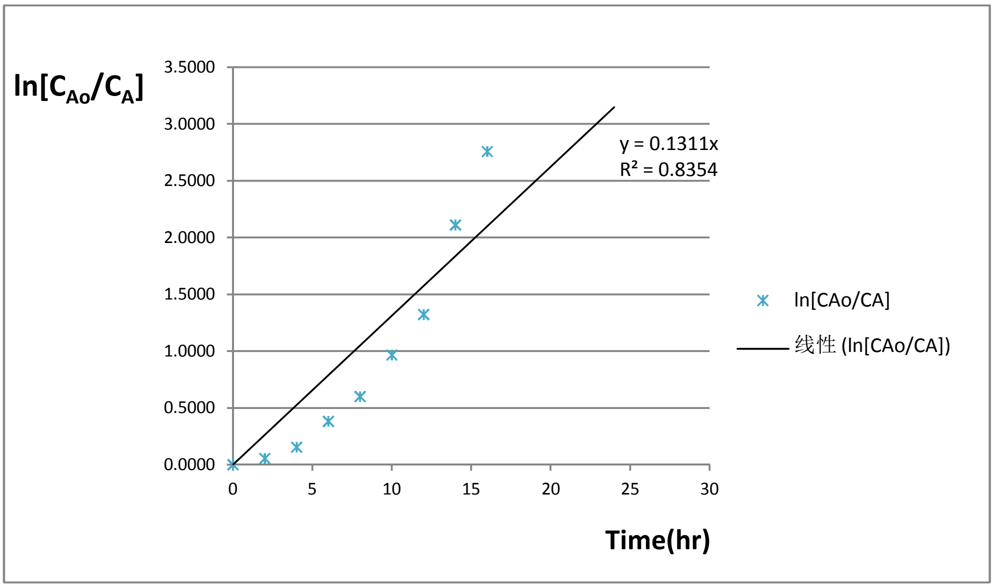

Fig. 16b. A plot of $\ln\left[\frac{C_{Ao}}{C_A}\right]$ against t for crude B at 50% addition of toluene.

From the graph in Fig.16a the breakage rate constant for Crude A is $k_c = 0.0012hr^{-1}$ and from Fig 4.16b, it can be seen that the breakage rate constant of crude B is $k_c = 0.1311hr^{-1}$. From the two values obtained, it can be clearly seen that the breakage rate constant of crude A is far less than that of crude B when subjected to the same condition of solvent addition. This implies that crude A formed a more stable emulsion than crude B.
To be able to predict the state of the water in crude oil emulsion formed beyond the study range, a model was developed. Using the first other rate equation in terms of conversion of the emulsified water A to resolved water R (X_A) as follows;
As obtained above in equation (5)

$$\ln\left[\frac{C_{Ao}}{C_A}\right] = k_c t$$

But
$C_A = (1 - X_A)C_{Ao}$ 7
$\frac{C_A}{C_{Ao}} = (1 - X_A)$ 8
Substituting in the above equation we have,
$\ln\left[\frac{1}{(1-X_A)}\right] = k_c t$ 9
Therefore, at any time t, the conversion of the emulsified water A to resolved water R can be determined and the concentration of the emulsified water can also be obtained using,

$$C_A = (1 - X_A)C_{Ao}$$

For instance, the state of the 50% addition of toluene on crude A sample which is a more stable emulsion can be predicted if the emulsion is allowed to stay for a week (168hr).
Where

$$\ln\left[\frac{1}{(1 - X_A)}\right] = k_c t$$

$\left[\frac{1}{(1-X_A)}\right] = e^{k_c t}$ 10

$$(1 - X_A) = e^{-k_c t}$$

$X_A = 1 - e^{-k_c t}$ 11
Therefore, for one week settlement

$$X_A = 1 - e^{-(0.0012*168)}$$
$$X_A = 0.7984$$

So the concentration of water retain in the emulsion for one week of settling will be

$$C_A = (1 - 0.7984)0.75$$
$$C_A = 0.1512$$

To evaluate the period it will take for 99% resolution of the emulsified water, where

$$t = \ln\left[\frac{1}{(1 - X_A)}\right]/k_c$$

$$t = \ln\left[\frac{1}{(1 - 0.99)}\right]/0.0012$$

$$t = 3837.6418\,hr = 159.9 days \approx 160 days$$

So it will take the emulsion formed with 50% addition of toluene in Crude A approximately 160 days to resolve 99% of the emulsified water.

4. Conclusion

The effect of asphaltene solvency on the stability of water-in-oil emulsion was investigated in this work using two Nigerian crude oil samples. Solvency was modified using different solvents, namely, aliphatic (heptanes), aromatic (toluene), and their blend (heptol); and varying aqueous phase pH. The results largely corroborates the literature assertion that the extent to which asphaltenes are solvated is the controlling factor in determining the surface active nature of these colloidal aggregates and thus emulsion stability. A robust model was developed for predicting amount of water resolved with time during demulsification by various solvents. Our findings would facilitate appropriate deployment of suitable demulsifiers in the treatment of water-in-oil emulsions and contribute to better management of the emulsions, particularly in the Nigerian oil industry.

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